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STUDY OF CONSTITUTION DIAGRAM ALUMINUM-TANTALUM

by

V.M. Glazov, M.V. Mal'tsev, Yu. D. Chistyakov

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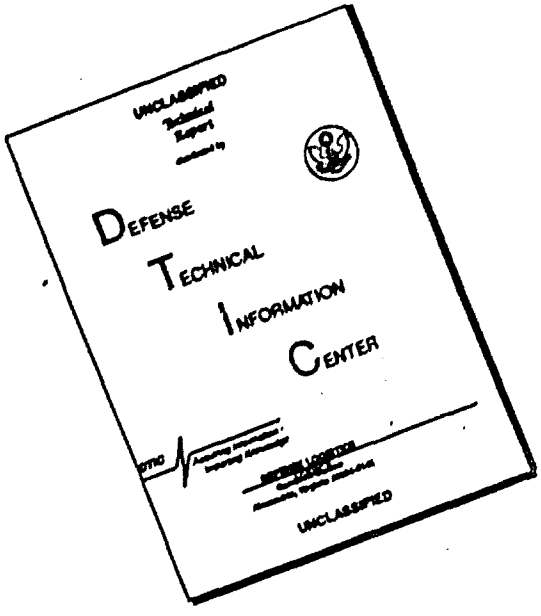

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*ye initially, after vowels, and after ъ, ь; e elsewhere.
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Russian	English	Russian	English	Russian	English
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cos	cos	ch	cosh	arc ch	$\cosh^{-1}$
tg	tan	th	tanh	arc th	$\tanh^{-1}$
ctg	cot	cth	coth	arc cth	$\coth^{-1}$
sec	sec	scn	sech	arc sch	sech^{-1}
cosec	csc	csch	csch	arc csch	csch^{-1}

Russian	English
rot	curl
lg	log

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STUDY OF CONSTITUTION DIAGRAM ALUMINUM-TANTALUM.

V. M. Glazov, M. V. Mal'tsev, Yu. D. Chistyakov (Moscow).

Alloys of aluminum with tantalum were for the first time obtained by aluminothermic method in 1868 by Moriniak. Later these alloys were studied in the works of Schirmeister (1915) and Brouwer (1938), moreover Brouwer established that tantalum with aluminum forms the chemical compound $TaAl_3$, which has tetragonal crystal lattice with parameters a=5.422 Å and c=8.536 Å [1].

However despite the fact that alloys of aluminum with tantalum long ago are obtained already, constitution diagram of this system is not studied until recently. In connection with the application of tantalum as the modifying additive in aluminum alloys [1] arose emergency in the construction of this diagram, without the knowledge by which it is not possible to give the correct explanation of the mechanism of the very process of the modification of primary grain. for this purpose was undertaken this work.

Preparation of alloys. As initial materials for the preparation of alloys they served: aluminum of the highest purity/finish (AB000) and metallic tantalum (99.5% Ta, 0.2% Nb, 0.1% Al and 0.2% of other admixtures/impurities). The investigated alloys from 0.01% to 2.5% were prepared with the dilution of initial alloy with 5.1% Ta,

obtained by the direct alloying of primary materials. During the manufacture of initial alloy tantalum was dissolved in the overheated to 1300° and machined by flux (45% KCl 45% NaCl and 10% K_3AlF_6) aluminum. Dissolution was continued for 1.5 hours. All alloys were prepared in the electric furnace, in the alundum crucibles. Were prepared alloys with content of 0.01, 0.03, 0.05, 0.1, 0.3, 0.4, 0.5, 0.6, 1.24, 2.5, 5.1% of tantalum.

Homogenization of alloys. For reducing the alloys into the state of equilibrium and for the equalization of the chemical composition the latter after the insignificant strain (to 30%) underwent prolonged homogenization for a week at a temperature of 500°C and subsequent in stages annealing at temperatures of 630°, 580°, 500°, 400°, 200°C for 40 hours at each temperature. Cooling from one to another temperature was manufactured with the furnace.

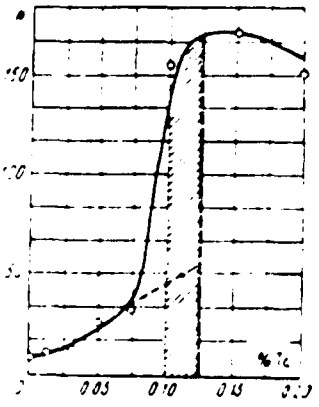

Fig. 1. Dependence of number of grains n, which fall on 1 cm^2 of area of large section, from % additive, tantalum; shaded region corresponds to beginning of precipitation of crystals of compound TaAl.

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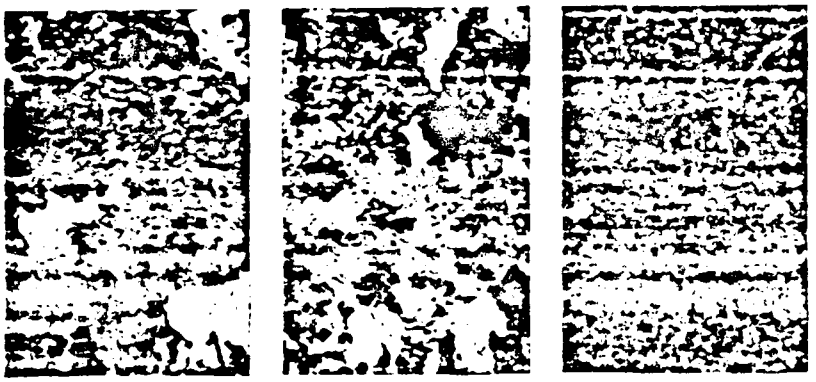

Fig. 2. Macrostructures of aluminum and some alloys of system aluminum- tantalum; a) aluminum AB000; b) aluminum +0.03% Ta; c) aluminum +0.15% Ta (full size).

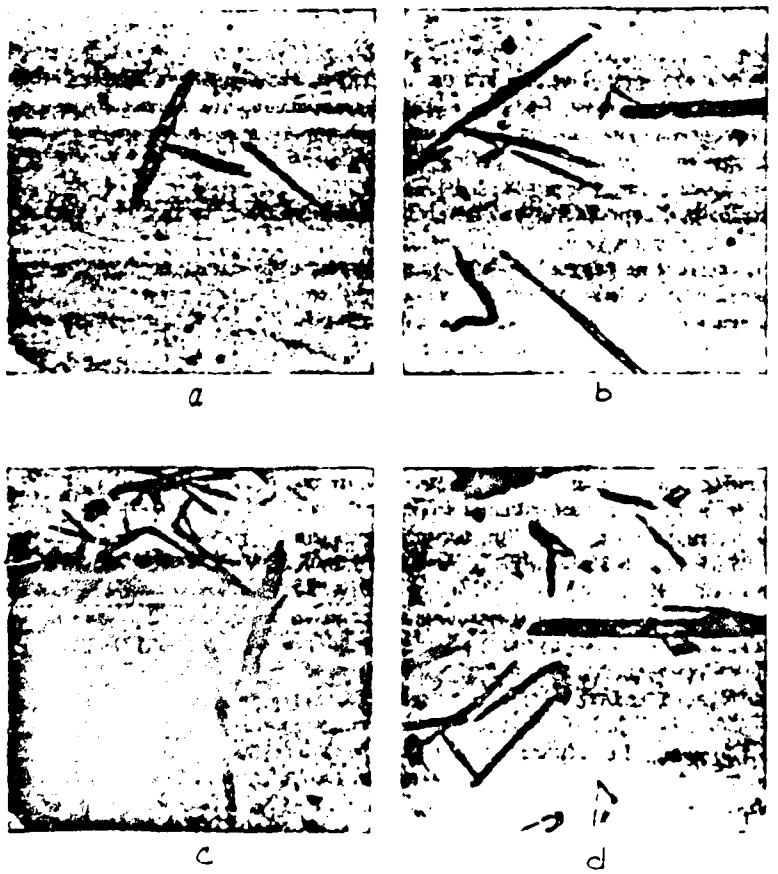

Fig. 3. Microstructures of some alloys of system aluminum-tantalum (electro-polishing, ×200); a) aluminum +0.3% Ta; b) aluminum +0.6% Ta; c) aluminum +1.24% Ta; d) aluminum +2.5% Ta.

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Chemical analysis. Chemical analysis of alloys was manufactured in the homogenized samples employing the following developed by us procedure[1].

FOOTNOTE [1]. During the determination of very small quantities of tantalum this procedure gives somewhat lowered results. ENDFOOTNOTE.

The batch of alloy in 1 g was dissolved in 20 cm³ of the mixture of acids (100 ml HNO_3, d=1.4 g/cm³, 300 ml HCl, d=1.19 g/cm³, 150 ml H_2SO_4, d=1.34 g/cm³, 450 ml of water). Dissolution was manufactured during the preheating to the precipitation of remainder/residue of tantalic acid. In this case aluminum goes into solution. Precipitate was washed clean and after calcination was weighed. With this precipitation method the part of tantalum goes into solution as a result of the partial dissolution of tantalic acid. For determining the remainder/residue to the filtrate it was added by 75-100 ml 1% solution of tannin (digallic acid). Tantalum remaining in the solution falls in the form of the organic compound of tantalum with the tannin. Precipitate was held for a day and after drying underwent calcination. After the calcination of upsettings it changes into the tantalum pentoxide Ta_2O_5. The results of the first and second

determination were totaled. Chemical-analysis data of alloys were taken as average/mean of fi e determinations.

Macro-and microstructure of alloys. Macrographic examination was manufactured in the poured samples, cut out from the ingots with different content of tantalum. For the determination of structure three reagents were used (Table 1).

The specimen was first etched and complex reagent 1, then it was clarified by solution of alkali and was washed then in solution of nitric acid for purpose of neutralization of effect/action of remainders/residues of alkali.

Fig. 2 gives photographs of large sections of some alloys. As follows from these photographs, the strong grinding of grain of aluminum is observed during the introduction of tantalum. For the quantitative estimation of the effect of grinding with a change in tantalum concentration the calculation of the grains, per unit of area was manufactured (cm^2). The results of calculation are integrated on the graph: the number of grains - % additive (Fig. 1).

This dependence has completely regular character. Initially in the addition of insignificant quantities of tantalum, considerable refining of the grain of aluminum it is not observed (Fig. 2b, Fig. 1 - initial section of curve). Then, on the achievement of the specific concentration of tantalum in the alloy, the sharp refining of grain

occurs (Fig. 2c). The analogous dependence of the number of grains to the square centimeter on the percentage of additive occurs in the systems aluminum-titanium, aluminum-zirconium, etc. [2].

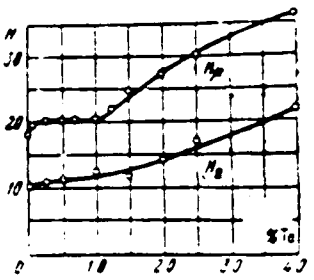

Fig. 4. Dependence of microhardness $H_к$ of crystals of solid solution and macrohardness $H_в$ on composition of alloys of system Al-Ta.

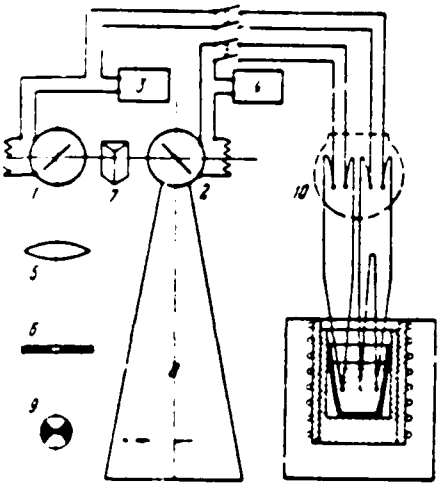

Fig. 5. Diagram of differential pyrometer of le Chatelier-Saladen; 1 and 2 - differential and simple galvanometers, 3 and 4 - resistance boxes, 5 - focusing lens, 6 - diaphragm, 7 - rotary prism, 8 - shield, 9 - light source, 10 - Dewar vessel with cold junctions by simple and differential thermocouples.

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During the comparison of these curves with the equilibrium constitution diagrams it turned out that the sharp refining of the

grain of aluminum occurs in them, beginning from the specific concentration, which corresponds to the beginning of precipitation of the primary crystals of chemical compound. As showed calculations and experiments, carried out by M. V. Mal'tsev (doctoral dissertation, Institute of nonferrous metals and gold im. M. I. Kalinin), the chemical compounds, which satisfy the rule of structural and dimensional conformity, are the most effective grinders (modifiers) of primary grain. In the case of $TiAl_3$, ZrAl, and $TaAl_3$, on one hand, and aluminum, on the other hand, this rule is fulfilled very well, moreover in the best way in the case of $TaAl_3$. during the imposition on the crystal face of the chemical compound TaAl, of the planes of cube the lattice of aluminum is observed a good coincidence of flat/plane atomic grids. Disagreement in the interatomic axial distances a composes 4.9%, and along axis/axle c - only 0.1%.

Thus, it is obtained, what plotting of curves (number of grains per unit of area - percentage of additive) is peculiar method of determining concentration, which corresponds to beginning of precipitation of crystals of chemical compound when, according to precomputations, crystal lattice of chemical compound is isomorphous to crystal lattice of primary grains crystallizing after it. The number of grains to the square centimeter - the percentage of additive for the system aluminum-tantalum, follows from the analysis of dependence that precipitation of the crystals of the chemical compound $TaAl_3$ occurs after the introduction of approximately 0.1-0.15% tantalum.

Microscopic analysis and measurements of microhardness of structural components and hardness were carried out for purpose of study of phase composition of alloys of system of aluminum-tantalum. For the microscopic analysis and the investigation of microhardness the ground joints were prepared by the method of electropolishing[1].

FOOTNOTE [1]. All samples to heat treatment under the given above conditions. ENDFOOTNOTE.

Investigations of microstructure showed that in alloys with content of tantalum approximately higher than 0.3% are present crystals of chemical compound $TaAl_3$. A quantity of these crystals is increased with an increase in the content of tantalum (Fig. 3a, b, c, d).

Structures, given in Fig. 3, are typical structures. Nothing other, qualitatively different from these structures, it was discovered during the most thorough investigation from 0.3% Ta to 5.1 of tantalum.

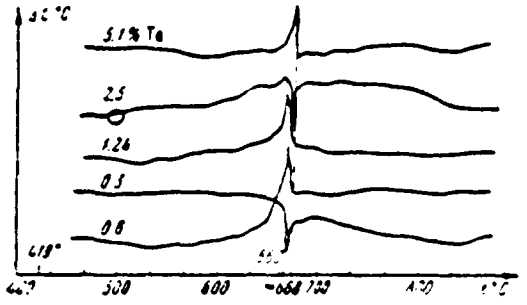

Fig. 6. Fig. 7.

Fig. 6. Cooling curves of some alloys of system Al-Ta.

Fig. 7. Change in pericd a of lattice of α-solid solution in dependence on tantalum concentration in alloy.

Table 1.

(1) № реактива	(2) Глицерин, мл	(3) HNO_3, мл	(4) HCl, мл	(5) HF, мл	$FeCl_3$,	(6) NaOH кристал.	(7) Вода, мл
1	18	49	75	1.5	9	—	—
2	—	—	—	—	—	20 г	1000
3	—	20	—	—	—	—	80

Key: (1). No. of reactant. (2). Glycerin, ml. (3). ml. (4). NaOH, crystallized. (5). Water, ml.

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The study of the microhardness of structural components was done on the same ground joints, as microscopic analysis. This investigation showed that the chemical compound has a microhardness 440-450 kgf/mm[1].

FOOTNOTE [1]. This value is, apparently rreduced as a result of the effect of soft backing/block. ENDFOOTNOTE.

The microhardness of the crystals of the solid solution in the dependence on the content of tantalum in the alloy is changed from 20 to 40 kg. The dependence of the microhardness of the crystals of solid solution and macrohardness on the composition of alloy is given in Fig. 4. Bend in the curve of microhardness answers, obviously, the maximum saturation of the solid solution of tantalum in aluminum at room temperature. In the curve of macrohardness this bend is expressed less clearly.

Thermal analysis. The thermal analysis of alloys was conducted on the differential pyrometer of Le Chatelier-Saladen with the recording of thermal curves by inertia method. The installation diagram is given in Fig. 5. Tool makes it possible to recover differences in temperatures between the junctions of the differential thermocouple of the order of several thousandths of a degree. For increasing the sensitivity of method the shield settled from the mirror of galvanometer at a distance of 2.5 meters, and the junctions of simple and differential thermocouples, greased by protective coating, were placed directly in the fusion/melt. The calibration of tool was manufactured according to melting points of the clean of zinc ($t_{пл}$ 419°C) and aluminum ($t_{пл}$ 660°C). Cooling was manufactured with the furnace, moreover crucible for the evenness of heating was placed into the massive copper cylinder. Fig. 6 gives the photocopies of cooling curves, obtained from some alloys. In the thermal curves all alloys, beginning with 0.3% Ta at one and the same temperature of 668-669°C, thermal effect is noted. Temperature constancy indicates the invariant character of transformation. In the interval of operating temperatures of 400-800°C accepted by us to recover the critical points, which correspond to primary crystallization, did not succeed. Evidently, liquidus line in this part of the diagram goes steeply upward and it falls outside the region of studied by us temperatures.

X-ray diffraction analysis[1].

FOOTNOTE [1]. Within this section in the work V. V. Glazov took part. ENDFOOTNOTE.

The X-ray diffraction analysis of alloys was conducted for the purpose of the establishment of the solubility of tantalum in solid aluminum. For this, as usual, was initially constructed the curve, which characterizes a change in the period of the lattice of solid solution with a change in the concentration of the dissolved element. For the construction by this curve were used the alloys[3] with content of 0.05, 0.1 and 0.2% Ta, hardened/tempered from temperature of 630°C after holding at this temperature for 20 hours.

FOOTNOTE [3]. These alloys, according to the data of microscopic analysis, are single-phase. ENDFOOTNOTE.

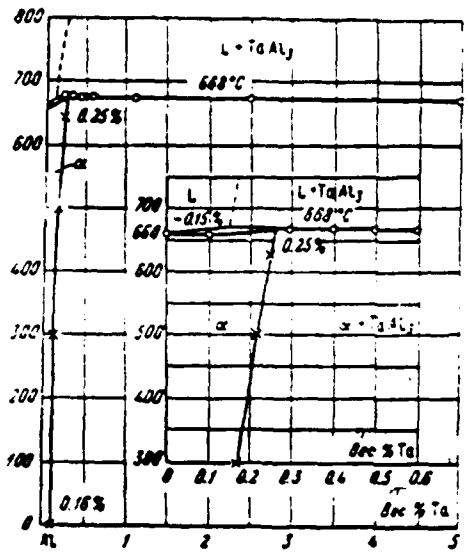

Fig. 8. Constitution diagram aluminum-tantalum (within it is represented angle of diagram on increased scale): o - data of thermal analysis, ×- data of X-ray diffraction analysis.

Key: (1). Weight % Ta.

Table 2. Solubility of tantalum in aluminum at different temperatures.

[illegible]	(1) Параметр решетки, Å	(2) Предельная растворимость
[illegible]	4.0447	0.24
[illegible]	4.0445	0.22
300	4.0440	0.18
[illegible]	4.0435	[illegible]

Key: (1). Lattice parameter, Å. (2). Critical solubility.

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(Alloys they underwent preliminarily prolonged homogenization, see above). For photographing of X-ray photographs of the alloys were filed the powders, which underwent reheating in copper ampules at a temperature of 630°C for two hours with their subsequent water quenching. X-ray photographs were removed/taken in the precise chambers/cameras of the type of Preston on the rays/beams of K-series of copper. The results of measuring the periods of lattice are given on the graph (Fig. 7). The concentration of the saturated solid solution at different temperatures was determined on the alloy, which contains 5.1% of tantalum. From this alloy, hardened/tempered from temperature of 630°C, were filed the powders and they were annealed at temperatures of 630°, 500° and 300°C. The results of investigation are represented in Table 2 and Fig. 7.

Construction of aluminum angle of constitution diagram of aluminum-tantalum. On the basis of the conducted investigations it is possible to give approximate drawing of the constitution diagram of aluminum with tantalum. The version of this diagram is given in Fig.

8. the type of diagram can be related to peritectic, since the temperature of invariant equilibrium lies/rests above temperature of the transformation of pure aluminum. Liquidus line on the diagram is plotted/applied by dotted line in view of the fact that in this work its accurate position is not established.

Conclusions. 1. It is established on the basis of microscopic and thermal analysis of alloys that in alloys aluminum-tantalum at temperature of 668-669°C occurs peritectic reaction $TaAl_3+L\rightarrow\alpha$.

2. It is roentgenographically established that tantalum to 0.25% is soluble in aluminum in solid state, moreover solubility insignificantly is reduced with decrease of temperature.

3. As a result of conducted investigations is given in the first approximation, outline of aluminum angle of constitution diagram aluminum-tantalum.

Submitted 26 Dec 1955.

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